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BOUNDARY MIGRATION AND DISAPPEARANCE  
OF VOIDS IN  $\alpha\text{-Al}_2\text{O}_3$  AT  $2000^\circ\text{C}$

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16. Abstract A series of photographs taken with Osaka University's high temperature 3MV electron microscope of $\alpha$ -Al <sub>2</sub> O <sub>3</sub> at 2000°C is presented. The dynamic study shows grain boundary migration in progress and demonstrates that disappearance of voids is controlled by boundary migration.			
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BOUNDARY MIGRATION AND DISAPPEARANCE  
OF VOIDS IN  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> AT 2000°C

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In general, ceramics are hard, heat resistant, corrosion resist-<sup>/634\*</sup>ant and have excellent insulating properties but their great fault is that they are brittle. This is due to the fact that most ceramics used for industrial purposes are made by sintering powders, resulting in countless voids and a weakening of grain boundaries. Trying to eliminate these problems is a constant problem in ceramics research but because of the minute structure, research with the electron microscope has become increasingly prominent in recent years and has given great results in the study of grain boundary structure through lattice images or the analysis of minute crystalline structures. To study these mechanical properties, however, as has been done for metals<sup>1)</sup>, the most effective method is "on-the-spot observation", or using a sample of test material with the necessary thickness to show the intrinsic qualities of the substance and directly observing the phenomena which occur under various conditions. With ceramics, however, it is necessary to bring the temperature in the microscope to 2000°C or more which leads to many problems in the development of such a device. At present, Osaka University Research Center for Ultra High Voltage Electron Microscopy has developed a 3MV microscope which can be heated to 2000°C<sup>2)</sup>, and is conducting dynamic studies of high temperature characteristics of ceramics and other materials with high melting points.

Using this apparatus, they are directly investigating the effects of MgO on the sintering of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> by comparing samples with and without this additive and are making direct observations of the dynamics of boundary migration and the disappearance of voids<sup>3)</sup>. The photographs are one example of those recorded on a VTR. The test material

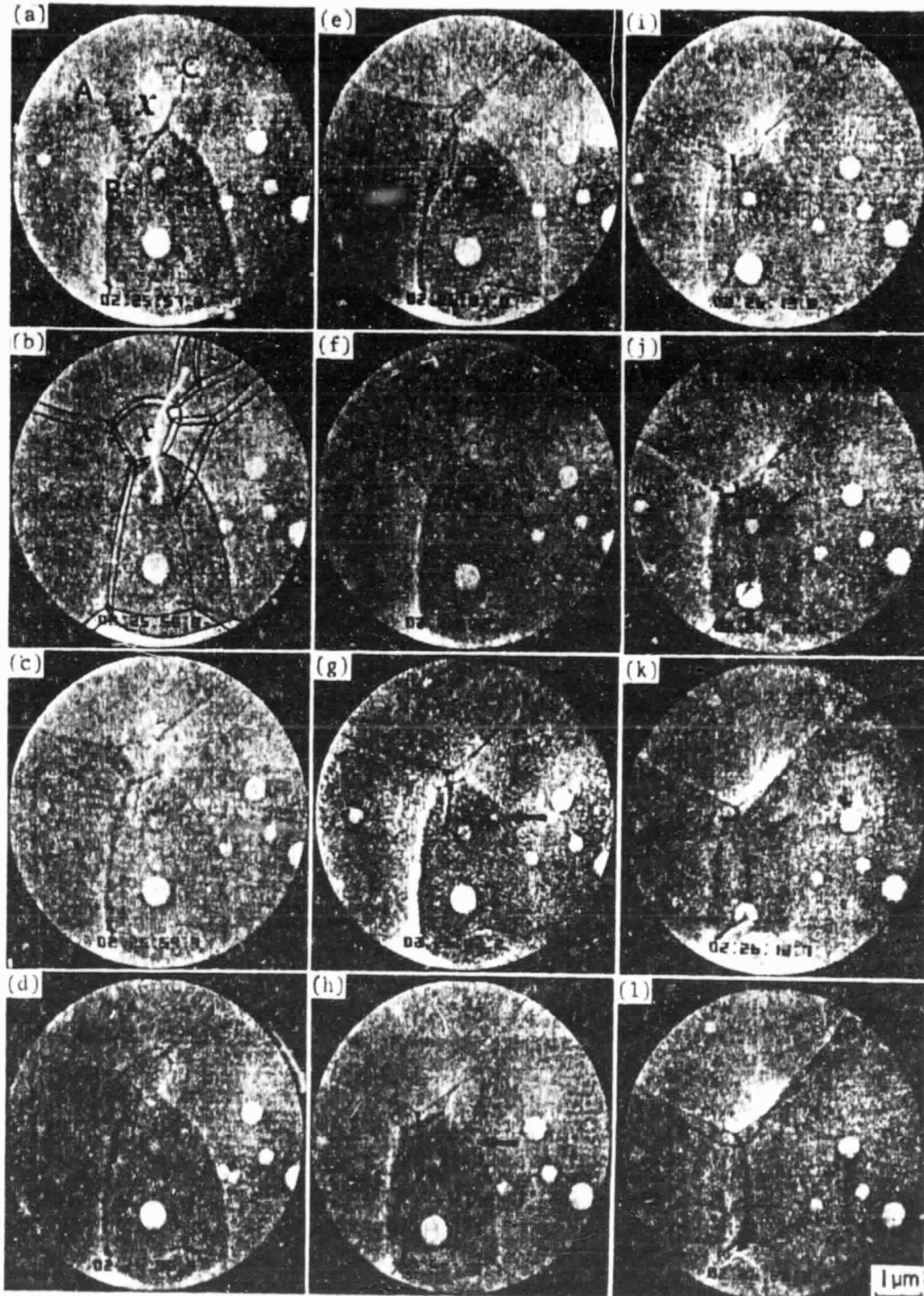
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\*Numbers in the margin indicate pagination in the foreign text.

was  $\alpha\text{-Al}_2\text{O}_3$  with 0.1wt% of MgO added and preliminary sintering done at  $1300^\circ\text{C}$ . Samples were then mechanically polished with diamond paste or ion etched to a thickness of several  $\mu\text{m}$ . At  $2000^\circ\text{C}$ , boundary migration becomes extensive and at the same time, voids suddenly disappear. The belt-like structures in the photographs are crystal grain boundaries formed after sintering. The white circular shapes are voids which remain in the system. The numbers printed on the bottom of each photograph are, from left to right, hour, minute, second, and 1/10 second. /635

At close to  $2000^\circ\text{C}$ , even in  $\alpha\text{-Al}_2\text{O}_3$ , grain boundary reactions begin to occur rapidly, starting at grain boundary triple overlap points and crystal grains surrounded by grain boundaries with high boundary energy suddenly disappear due to the cooperative migration of those grain boundaries. For example, the particle marked with an x in frame (a) contracts in a few seconds, (b)-(e), and in frame (f) has completely disappeared. The speed of migration of the voids, however, even at  $2000^\circ\text{C}$ , becomes markedly slower and in frames (a)-(l), their positions are almost unchanged and very few of these escape to the surface. When these voids which are stable against heat meet a grain boundary, they are suddenly absorbed in the grain boundary and disappear. For example, in frame (a), underneath the x mark can be seen a crystal grain with 3 voids - large, medium, and small. The boundaries of this grain are moving quickly, as can be seen in the pictures. First, the grain boundary on the left moves and when it meets the small void ( $\uparrow$ ) in frame (g), it quickly disappears (h). Next, the medium-sized void meets a grain boundary moving from the right (j) and quickly disappears. The largest void meets a grain boundary triple overlap point migrating from the bottom and is absorbed by it in frames (j)-(l). In other words, it can be seen that disappearance of voids is controlled by boundary migration. Thus, using the ultra high voltage electron microscope, it is possible to conduct dynamic studies of the high temperature properties of ceramics using lattice imperfections as a scale. At present, it is becoming possible to conduct tension deformation at  $2000^\circ\text{C}$  and great results are expected in the future. /636

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